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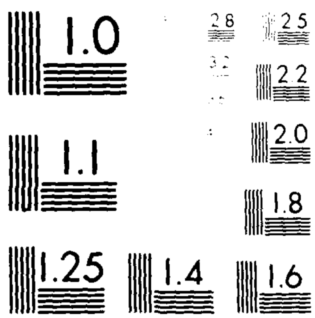

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Isotope Effect in Gas-Surface Vibrational Energy Transfer.

Cyclopropane and Cyclopropane-d_6 Isomerization by the VEM Technique

by W. Yuan, R. Tosa, K.-J. Chao, and B. S. Rabinovitch

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Technical Report No. NR092-549-TR22

Contract N00014-75-C-0690, NR-092-549

October 20, 1981

Prepared for Publication in
Chemical Physics Letters

OFFICE OF NAVAL RESEARCH
Department of the Navy
Code 473
800 N. Quincy
Arlington, VA 22217

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER: NR092-549-TR22

2. GOVT ACCESSION NO.: AD-A107365

3. RECIPIENT'S CATALOG NUMBER:

4. TITLE (and Subtitle): Isotope Effect in Gas-Surface Vibrational Energy Transfer. Cyclopropane and Cyclopropane-d_6 Isomerization by the VEM Technique.

5. TYPE OF REPORT & PERIOD COVERED: Technical

6. PERFORMING ORG. REPORT NUMBER:

7. AUTHOR(s): W. Yuan, R. Tosa, K.-J. Chao [redacted] B. S. Rabinovitch

8. CONTRACT OR GRANT NUMBER(s): N00014-75-C-0690 NR092-549

9. PERFORMING ORGANIZATION NAME AND ADDRESS:
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Department of Chemistry BG-10
University of Washington
Seattle, WA 98195

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS:

11. CONTROLLING OFFICE NAME AND ADDRESS:
Office of Naval Research, Code 743
Department of the Navy
800 N. Quincy
Arlington, VA 22217

12. REPORT DATE: Octo[redacted]981

13. NUMBER OF PAGES: 18

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office):

15. SECURITY CLASS. (of this report): Unclassified

15a. DECLASSIFICATION DOWNGRADING SCHEDULE:

16. DISTRIBUTION STATEMENT (of this Report)

This document has been approved for public release; its distribution is unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

Prepared for publication in Chem. Phys. Lett.

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Accommodation	Isotope Effects
Cyclopropane	Surfaces
Energy Transfer	Unimolecular Reaction
Gases	Vibrational Relaxation
High Temperature	

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Study of varying molecular structure on the efficiency of vibrational energy transfer between molecules and a hot surface has been extended to the pair, cyclopropane/cyclopropane-d_6. The latter is less efficient in energy loss, as corresponds to the greater vibrational eigenstate density. Conversely, the probability of energy up-transitions is enhanced; in thermal low pressure unimolecular reactions this corresponds to the inverse statistical weight secondary isotope effect.

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Isotope Effect in Gas-Surface Vibrational Energy Transfer. Cyclopropane and Cyclopropane-d_6 Isomerization by the VEM Technique[+]

by W. Yuan,[#] R. Tosa, K-J. Chao[#] and B. S. Rabinovitch

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Abstract

The study of the effect of varying molecular structure upon the efficiency of vibrational energy transfer between initially cold molecules and a hot surface has been extended to the reaction pair, cyclopropane/cyclopropane-d_6. The latter member is somewhat less efficient in energy <u>loss</u>, as corresponds to the greater vibrational eigenstate density. Conversely, the probability of energy up-transitions is enhanced; in thermal low pressure unimolecular reactions this corresponds to the inverse statistical weight secondary isotope effect. Comparison is made with other molecules.

Introduction

The Variable Encounter Method (VEM) has been shown to be a useful technique[1,2] for the study of gas-wall vibrational energy transfer in the transient regime of a reacting system. In this method, an initial vibrationally-cold ensemble of molecules is excited into a vibrationally-hot distribution by a known, and variable sequence of successive collisions with a hot surface. To date, we have used seasoned fused-quartz surfaces. The rate of vibrational excitation, i.e. the rate of transient relaxation, is derived from the rate of unimolecular reaction.

VEM has been applied to the study of a number of substrate hydrocarbon species, such as the reactions of cyclopropane,[3] cyclopropane-d_2,[1] cyclobutane,[2,3] methylcyclopropane,[4] and cyclobutene.[5,6] These molecules have similar critical thresholds ($E_0 \sim 60$ kcal mole^{-1}), except for cyclobutene for which the value of E_0 is approximately one-half. As described in refs. 1-4, the efficiency of energy relaxation decreases with increasing temperature; also, the average amount of energy transferred per collision from a molecule to the wall, i.e. the size of an energy down-jump, $\langle \Delta E' \rangle$, declines with increasing molecular complexity (while a concomitant increase in the probability of up-transitions also occurs). It was pointed out[4] on the basis of a quasi-statistical accommodation model[7] that such behavior should follow with increase in the number of, and reduction of the frequencies of the vibrational modes of the substrate molecules.

To clarify further the effect of vibrational frequency pattern and vibrational energy level density on the energy relaxation process, and especially to simplify the comparison, a study of a simple isotopic pair, cyclopropane and cyclopropane-d_6 seemed desirable. Furthermore, in order to reduce the effect of experimental vagaries, it was desirable to study both species simultaneously in the same reactor and thus under identical conditions of seasoning and of

surface. The isomerization reaction has been shown previously to be free from surface catalytic effects under our conditions.[1,3]

The isotope effect and reaction parameters for this pair has previously been studied in a homogeneous steady-state thermal system over a range of pressures and degree of fall-off.[8] The activation energy for D-reaction is higher ($\sim$ 1.4 kcal mole^{-1}) than for H-reaction, but it was found that the quantum statistical secondary isotope effect overtakes the primary isotope effect as the second order, low-pressure region is approached. In the low-pressure regime, the reaction coordinate is, of course, simply collisional energy transfer and the low pressure thermal unimolecular rate constant is the inverse of the <u>steady state</u> mean first passage time,[9] $k_o = 1/\bar{t}_{fp}$.

Experimental

The apparatus used in this study was similar to that of previous VEM studies. The reactor vessel consisted of a 1-liter spherical quartz reservoir flask with two cylindrical reactor fingers blown on. Each finger had an inside diameter of 4 cm and either could be heated independently with a stainless steel block furnace. For finger lengths of $\sim$ 5 cm and $\sim$ 14 cm, the average numbers of sequential collisions, m, made by a molecule after entering the reactor and prior to returning to the reservoir (termed an encounter), was 5.0 and 14.4, respectively. Reaction temperature was varied from 876°K to 1060°K. Reactor temperature was measured with use of five chromel-alumel thermocouples cemented to the outside of the reactor finger. The temperature variation during the run time was ± 2°C, and that along the finger length was ± 5°C. The temperature of the reservoir flask wall (which fell in the range 100° - 150°C) was measured at several points, and being in the Knudsen region, the temperature of the reservoir gas was simply averaged with suitable weighting for surface area (i.e. by $\sin 2\theta$, where θ is the angular deviation from the perpendicular to the entrance area of the reactor finger).

Cyclopropane (Matheson, 99.9% with 0.08% propene as the major impurity) and cyclopropane-d_6 (Merck, 98% isotopic purity) were degassed and stored in a reactant bulb as a 1:1 molar ratio gas mixture. Before a run, the reactor was evacuated to approximately 10^{-6} torr. Reaction run pressures were in the range $1\text{-}2 \times 10^{-4}$ torr. Reaction time varied from several minutes to ten hours, depending on the reaction temperature. The reaction percentage varied between 5% and 40%. At the end of a run, the reaction mixture was expanded from the reactor into a liquid nitrogen trap. Separation of the reaction products was made at 0°C on a 20 m column of 3 mm nylon tubing packed with a 25% silver nitrate-ethylene glycol solution on 40-60 mesh Chromosorb P. A very short

column of mineral oil on Chromosorb P followed in order to protect the FID detector from contamination due to bleeding of ethylene glycol. Standard mixtures of light and heavy cyclopropanes and propenes were used for the calibration of peak areas. No side products were observed.

Results and Discussion

The isomerization reaction of cyclopropane and cyclopropane-d_6 to propene and propene-d_6, respectively, follow the first order law.[2] The apparent rate constants are summarized in Table 1. Each tabulation is the average of from 2 to 4 determinations from separate runs. As Figure 1 shows, the Arrhenius relation (which no longer has a simple meaning here) was satisfied for this limited temperature range. The apparent activation energies calculated from Figure 1 are given in Table 2 and agree well with earlier[1] measurements made in a different reactor.

The experimental ratios of production of the products, $R(D_6/H_6)$, were corrected for the differential numbers of collisions with the reactor, i.e. by the inverse ratio of the square roots of the molecular masses. The values are shown in Table 3 and plotted in Figure 2.

The average probability for isomerization per collision with the hot wall, $\bar{P}_c(m)$, was calculated from the apparent first order rate constants and the known reactor dimensions. The results are shown in Table 4 and Figure 3. There appears to be fairly good agreement between our $\bar{P}_c(m)$ values for cyclopropane and those of earlier measurements.[3] Unfortunately, the values of m in the earlier work were m = 2.6, 8.5 and 27.2 so that a direct comparison is not easily made; however, the present values fall in correct juxtaposition in Fig. 1 of ref. 3, relative to $\bar{P}_c(m)$ values plotted vs T(K) for the several m-values of the earlier work.

In order to fit the ratio data of Fig. 2 to theoretical stochastic calculations of the kind described in refs. 1-3, we have adopted the Gaussian form for the transition probability distribution described in ref. 3. The values for $\langle\Delta E'\rangle$, the average amount of energy transferred from a hot cyclopropane molecule by collision with the wall, that fit the data are $\langle\Delta E'\rangle$ (cm^{-1}) = 2400 (900 K); 2100 (1000 K) and 2000 (1100 K); the values given in ref. 3 are 2500,

2170, and 2040, respectively, in good agreement. The value of $\langle \Delta E' \rangle^D$ for cyclopropane-d_6 required to fit the experimental ratios in the m = 5.0 reactor are given in Table 5. The values are less than those for light cyclopropane and decrease with increasing temperature as has been found in earlier work.[1-5,10] These values of $\langle \Delta E' \rangle^D$ were then utilized to calculate predicted values of $R(D_6/H_6)$ for m = 14.4. These are shown in Fig. 2 as X. The values are low but in fair accord with the experimental m = 14.4 curves. The required fit values of $\langle \Delta E' \rangle^D$ to give exact concordance with the latter experiments are also given in Table 5 and differ only modestly from the m = 5 values. This signifies the near-reproducibility of our seasoned surfaces for present purposes.

A listing of various substrate molecules is provided in Table 6, together with the values of $\langle \Delta E' \rangle$ measured at various temperatures. It is evident that $\langle \Delta E' \rangle$ decreases with increase of molecular complexity and/or decrease in vibrational frequencies. At the same time, there is an increase in the value of $\bar{P}_c(m)$. The latter effect was characterized a number of years ago, as it appears in thermal unimolecular low pressure reactions for the case of replacement of H by D, as a rate enhancement due to an inverse statistical weight secondary isotope effect.[8,11,12] For the general case, such enhancement of low pressure thermal rate, due simply to change (increase) in vibrational eigenstate density of the molecule with increase in its molecular complexity, has been termed a generalized quantum statistical weight effect:[13] the centroid of the Boltzmann distribution shifts to higher energies, and the probability of energy up-transitions to the region of E_0 is enhanced. The inverse decline in $\langle \Delta E' \rangle$ also follows on the basis of a (quasi-)statistical energy transfer model[7] as the effective heat capacity of the molecular heat sink increases.

The decline in $\langle \Delta E' \rangle$ with rise of temperature accords with our earlier measurements and explanations[1-4,14] and follows from a quasi-statistical accommodation model, especially as limited by decreased surface residence time at higher temperatures.[7,14]

Table 1. Apparent rate constants for isomerization

$\underline{m}$	$\underline{T(K)}$	$10^6\ k_H\ (sec^{-1})$	$10^6\ k_D\ (sec^{-1})$
5.0	910	5.1 (0.7)[a]	5.1 (0.7)
	970	24.0 (2.7)	25.5 (2.5)
	1044	113 (8)	121 (8)
14.4	876	26.8 (0.3)	35.4 (0.4)
	879	25.5 (1.6)	33.6 (3.1)
	945	175 (4)	242 (4)
	1053	2360 (40)	3340 (10)
	1063	2810 (110)	3950 (60)

a) Standard deviation of an individual measurement

Table 2. Arrhenius activation energies for isomerization of cyclopropane.

$\underline{m}$	E_H(kcal mole^{-1})	E_D(kcal mole^{-1}
5.0	42.7 (42.5)[a]	44.0
14.4	46.5 (50.0)[b]	47.0

a) Value in ref. 1 fcr cyclopropane-d_2, m = 5.0

b) Value in ref. 1 for m = 22.

Table 3. The product ratios $R(D_6/H_6)$ for propene-d_6/propene[a]

m	T(K)	$R(D_6/H_6)$	
5.0	910	1.09	
		1.11	
		1.06	
		1.04	Av. 1.08 ± 0.02[b]
	970	1.18	
		1.09	
		1.11	
		1.15	Av. 1.13 ± 0.03
	1044	1.10	
		1.14	
		1.18	
		1.16	Av. 1.15 ± 0.02
14.4	876	1.40	
		1.43	
		1.41	Av. 1.41 ± 0.02
	879	1.39	
		1.43	Av. 1.41
	945	1.49	
		1.49	
		1.47	Av. 1.48 ± 0.01
	1053	1.53	
		1.51	Av. 1.52
	1063	1.54	
		1.47	Av. 1.51

a) Products brought to the same collision basis

b) Standard deviation of the mean

Table 4. Experimental values of $\bar{P}_c(m)$

m	T(K)	$10^8\ \bar{P}_c^H(m)$	$10^8 \bar{P}_c^D(m)$	$\bar{P}_c^D/\bar{P}_c^H$
5.0	910	1.35 (0.18)[a]	1.44 (0.18)	1.07
	970	6.18 (0.70)	7.02 (0.69)	1.14
	1044	27.5 (2.0)	31.6 (2.1)	1.15
14.4	876	2.50 (0.03)	3.53 (0.04)	1.41
	879	2.38 (0.15)	3.35 (0.31)	1.41
	945	15.8 (0.03)	23.4 (0.4)	1.48
	1053	202 (3)	306 (3)	1.51
	1063	241 (9)	362 (6)	1.50

a) Standard deviation of an individual measurement.

Table 5. Values of R(D/H) and $\langle \Delta E' \rangle^D (cm^{-1})$

T(K)	R(D/H)[a]	$\langle \Delta E' \rangle^D (cm^{-1})$
900	1.07	1980[a], 2040[b]
1000	1.14	1850[a], 1890[b]
1100	1.18[c]	1800[a,c], 1860[b]

a) from fit to m = 5.0 ratio data

b) from fit to m = 14.4 ratio data

c) Value obtained by extrapolation to permit comparison with ref 3.

Table 6. Comparison of $\langle\Delta E'\rangle_d(cm^{-1})$ and $\bar{P}_c(m)$ for Different Molecules

Molecule	E_o/kcal	m	$\bar{P}_c(m)\times10^8$			Model	$\langle\Delta E'\rangle_d(cm^{-1})$			Ref.
			900K	1000K	1110K		900K	1000K	1100K	
Cyclopropane	64	5.0	1.0	11.3	68	G	2400	2100	2000	this work
		14.4	4.8	62	540					
		8.5	4.5	37	260		2500	2170	2040	3
		27.2	5.9	69	660					
Cyclopropane-d_6	65.5	5.0	1.7	13.4	78	G	2040	1890	1860	this work
		14.4	6.7	89	800					
Cyclobutene	33					G	2150 (764K)			6
Cyclobutane	63	8.5	7.6	77	400	EXP	1800	1600	1480	2
		27.2	18.0	174	1000	G	2125	1925	1875	
Methylcyclo-propane	61	5.6	8.2	85	630	EXP	1550	1440	1415	4
		20	145	2200	15200					

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+ Work supported by the Office of Naval Research

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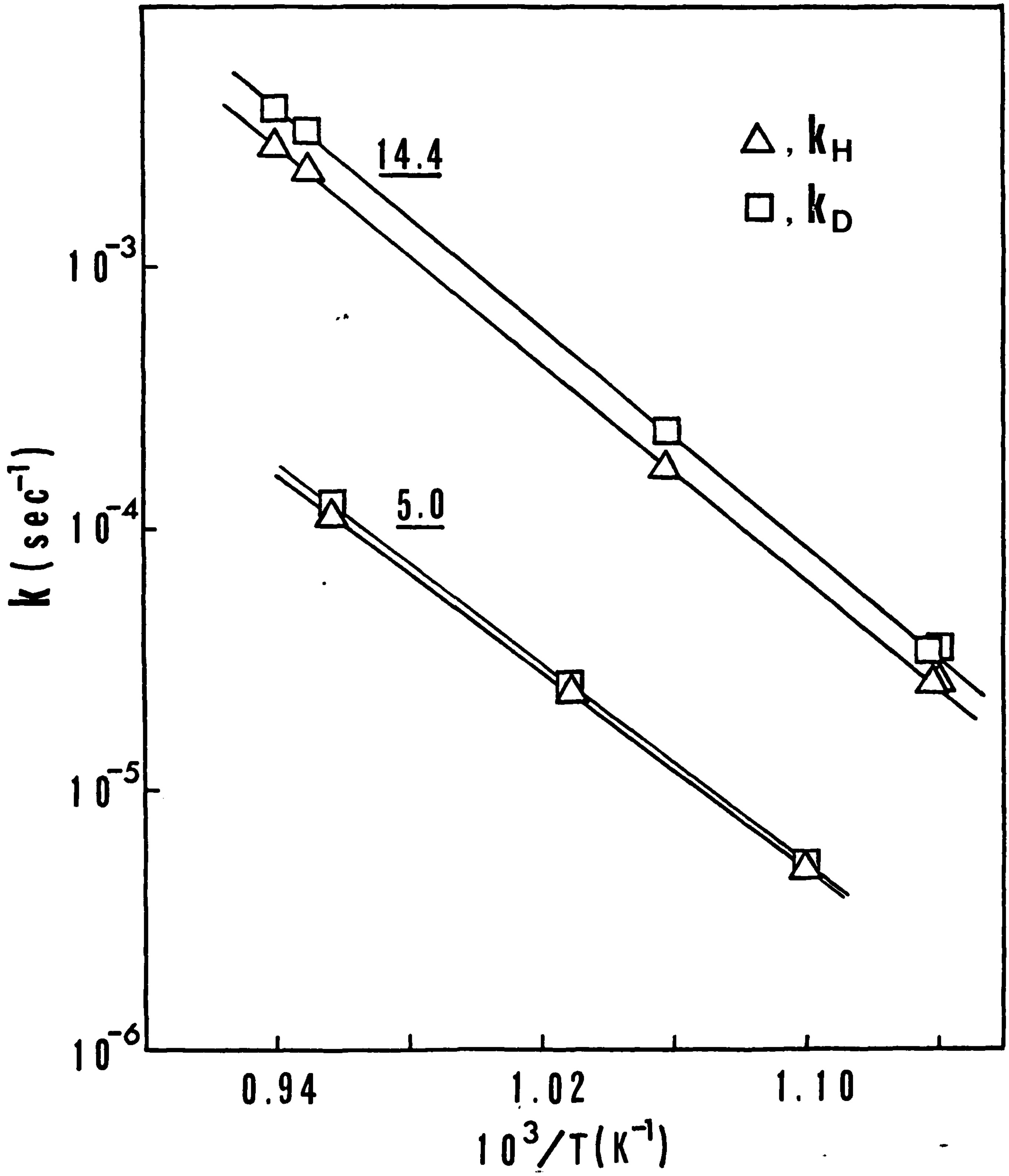
Figure Captions

Fig. 1 Arrhenius plots of log k_H, $\triangle$, and log k_D, $\square$, versus 1/T for the m = 5.0 and m = 14.4 reactors.

Fig. 2 Plot of $R(D_6/H_6)$ versus T for the m = 5.0, $\triangle$, and m = 14.4, $\square$, reactors. The values of $\langle \Delta E' \rangle$ that fit the data in the m = 5 reactor give the predicted curve (shown by the dashed line) for the m = 14.4 reactor.

Fig. 3 Plot of $P_c(m)$ versus T(K) for cyclopropane, $\triangle$, and cyclopropane-d_6, $\square$, for the m = 5.0 and m = 14.4 reactors.



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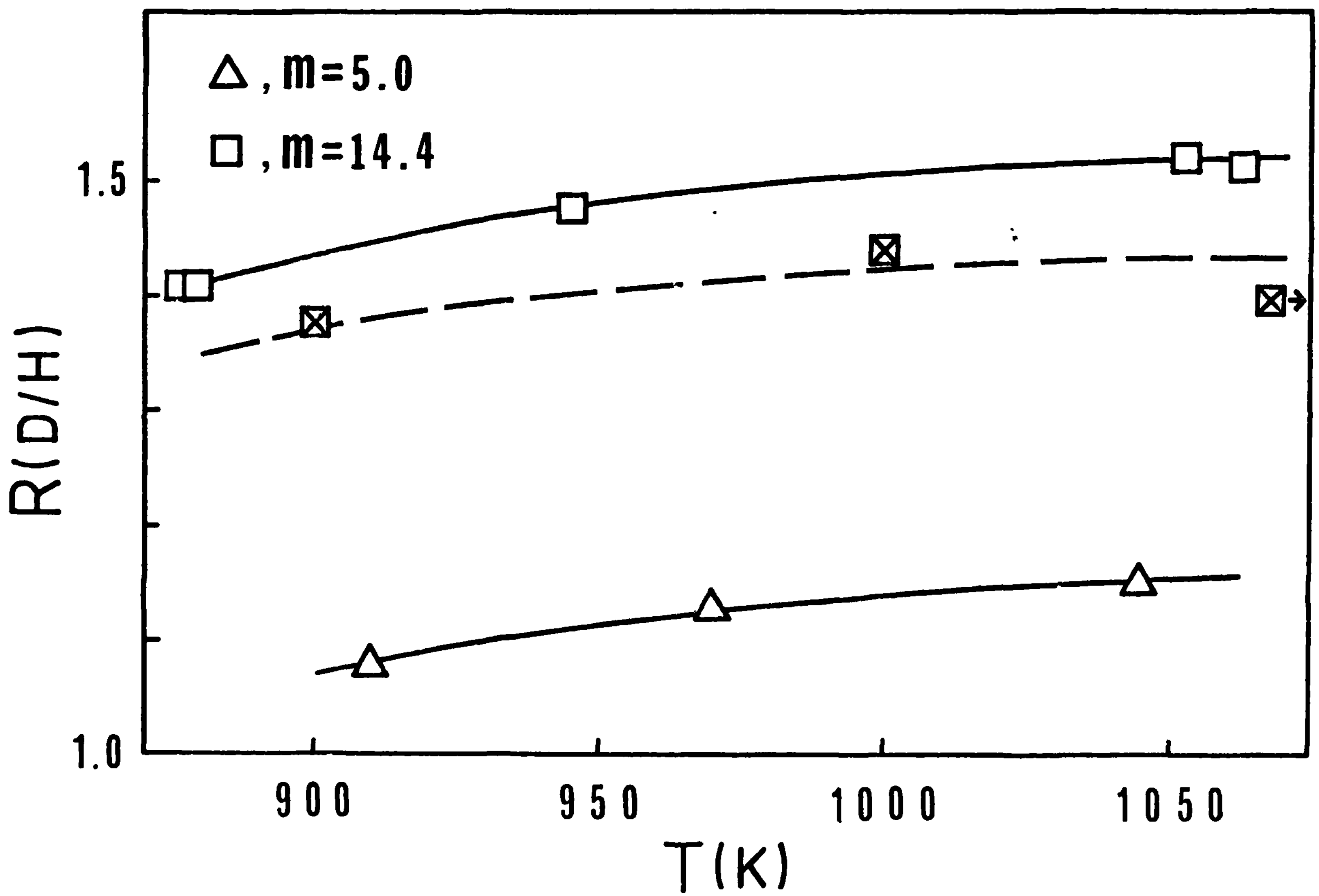

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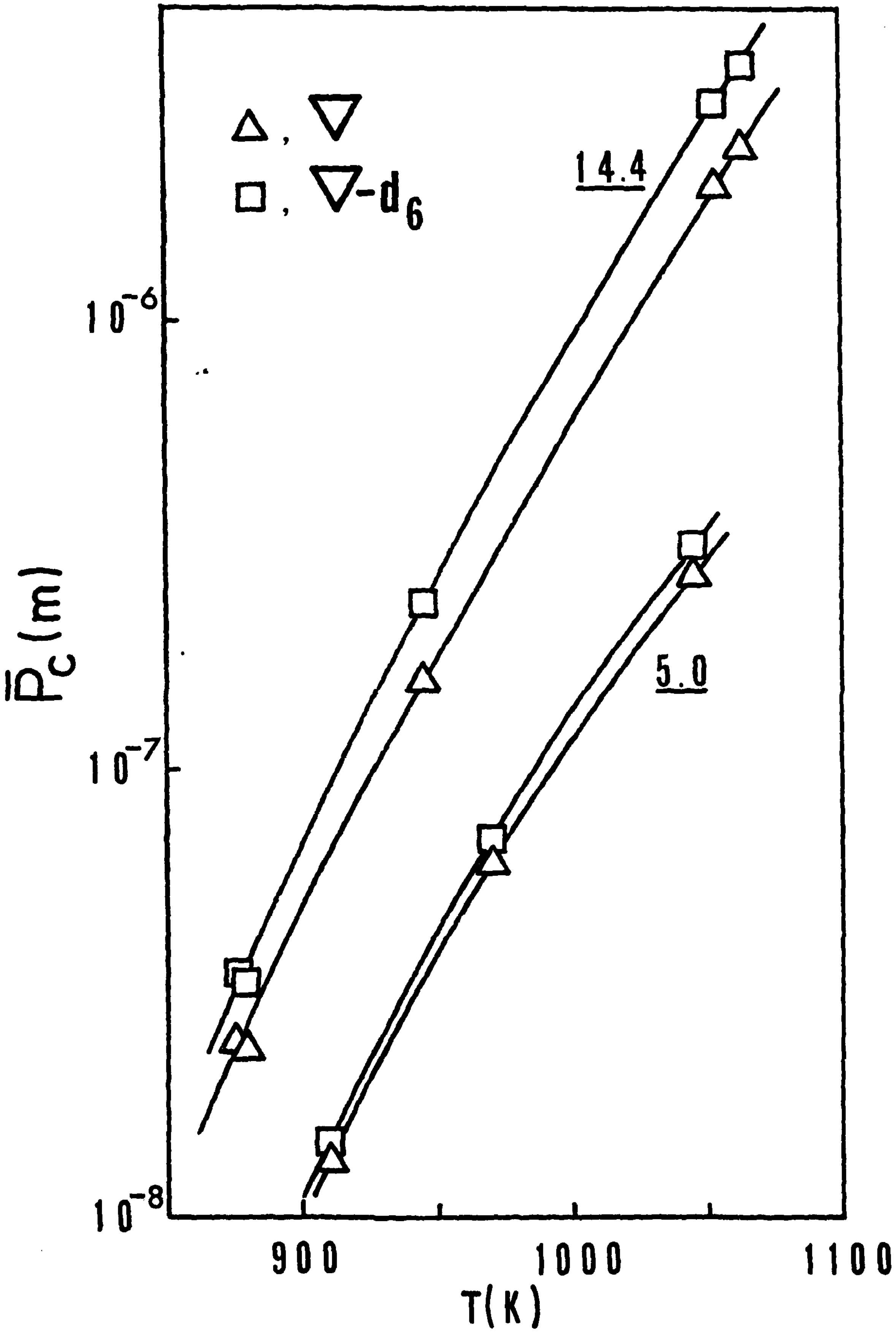

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